

**Compendium of Methods
for the Determination of
Inorganic Compounds
in Ambient Air**

Compendium Method IO-3.7

**DETERMINATION OF METALS
IN AMBIENT PARTICULATE
MATTER USING
NEUTRON ACTIVATION ANALYSIS
(NAA) GAMMA SPECTROMETRY**

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Method IO-3.7
Determination of Metals in Ambient Particulate Matter Using
Neutron Activation Analysis (NAA) Gamma Spectrometry

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Chapter IO-3
CHEMICAL SPECIES ANALYSIS
OF FILTER-COLLECTED SPM

Method IO-3.7
DETERMINATION OF METALS IN AMBIENT PARTICULATE MATTER USING
NEUTRON ACTIVATION ANALYSIS (NAA) GAMMA SPECTROMETRY

1. Scope

1.1 The area of toxic air pollutants has been the subject of interest and concern for many years. Recently the use of receptor models has resolved the elemental composition of atmospheric aerosol into components related to emission sources. The assessment of human health impacts resulting in major decisions on control actions by federal, state, and local governments is based on these data. Accurate measures of toxic air pollutants at trace levels are essential to proper assessments.

1.2 Suspended particulate matter (SPM) in air generally is a complex, multi-phase system of all airborne solid and low vapor pressure liquified particles having aerodynamic particle sizes from below 0.01-100 μm and larger. Historically, SPM measurement has concentrated on total suspended particulates (TSP), with no preference to size selection.

1.3 The most commonly used device for sampling TSP in ambient air is the high volume (hi-vol) sampler, which consists essentially of a blower and a filter and usually operates in a standard shelter to collect a 24-h sample. The sample is weighed to determine the concentration of TSP and is usually analyzed chemically to determine the concentration of various inorganic compounds. The hi-vol is considered a reliable instrument for collecting TSP in ambient air. When EPA first regulated TSP, the national ambient air quality standard (NAAQS) was stated in terms of SPM with aerodynamic particle sizes of $< 100 \mu\text{m}$ captured on a filter as defined by the hi-vol TSP sampler. Therefore, the hi-vol TSP sampler was the reference method. The method is codified in 40 CFR 50, Appendix B.

1.4 More recently, research on the health effects of TSP in ambient air has focused increasingly on those particles that can be inhaled into the respiratory system, i.e., particles of aerodynamic diameter of $< 10 \mu\text{m}$. These particles are referred to as PM_{10} . The health community generally recognizes that these particles may cause significant, adverse health effects. Therefore, the primary NAAQS for SPM is now stated in terms of PM_{10} rather than TSP. The reference method for PM_{10} is codified in 40 CFR 50, Appendix J, and specifies a measurement principle based on extracting an ambient air sample with a powered sampler that incorporates inertial separation of PM_{10} size range particles and collection of these particles on a filter for a 24-h period. Again, the sample is weighed to determine PM_{10} concentration and is usually analyzed chemically to determine the concentration of various inorganic compounds.

1.5 Current research strongly suggests that atmospheric particles commonly occur in two distinct modes: the fine ($< 2.5 \mu\text{m}$) mode and the coarse (2.5-10.0 μm) mode. The fine or accumulation mode (also termed the respirable particulate matter) is attributed to the growth of particles from the gas phase and subsequent agglomeration, whereas the coarse mode is made of mechanically abraded or ground particles. Because of their initially gaseous origin, the fine range of particle sizes includes inorganic ions such as sulfate, nitrate, and ammonia as well as combustion-form carbon, organic aerosols, metals, and other combustion products. Coarse particles, on the other hand, normally consist of finely divided minerals such as oxides of aluminum silicate, iron, calcium, and potassium.

1.6 Airborne particulate materials retained on a sampling filter, whether TSP or PM₁₀ size fractions, may be examined by a variety of analytical methods. This method describes the procedures for the neutron activation analysis (NAA) technique. The NAA method provides analytical procedures for determining percent, ppt, ppm, or ppb levels of 40-50 elements that might be captured on typical glass fiber filters used in hi-vol and dichotomous sampling devices. The NAA is not matrix dependent and is therefore highly applicable for elemental analysis of a broad spectrum of particulate material. A summary of elements for which NAA is a suitable analysis technique is provided in Table 1.

1.7 NAA should be performed by nuclear applications engineers, radiochemists, and inorganic chemists with related nuclear expertise. A minimum of 2-year's experience with NAA (of all matrices of interest) is recommended.

2. Applicable Documents

2.1 ASTM Documents

- D4096 *Application of High Volume Sample Method For Collection and Mass Determination of Airborne Particulate Matter.*
- D1356 *Definition of Terms Related to Atmospheric Sampling and Analysis.*
- D1357 *Practice For Planning the Sampling of the Ambient Atmosphere.*
- D2986 *Method for Evaluation of Air Assay Media by the Monodisperse DOP (Diocetyl Pthalate) Smoke Test.*

2.2 U.S. Government Documents

- STP598 Calibration in Air Monitoring
- U.S. Environmental Protection Agency, *Quality Assurance Handbook for Air Pollution Measurement Systems, Volume I: A Field Guide for Environmental Quality Assurance*, EPA-600/R-94/038a.
- U.S. Environmental Protection Agency, *Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II: Ambient Air Specific Methods (Interim Edition)*, EPA-600/R-94/038b.
- "Reference Method for the Determination of Particulate Matter in the Atmosphere," *Code of Federal Regulations*, 40 CFR 50, Appendix B.
- "Reference Method for the Determination of Particulate Matter in the Atmosphere (High Volume Method)," *Code of Federal Regulations*, 40 CFR 50, Appendix J.
- "Reference Method for the Determination of Particulate Matter in the Atmosphere (PM₁₀ Method)," *Code of Federal Regulations*, 40 CFR 50, Appendix J.
- "1978 Reference Method for the Determination of Lead in Suspended Particulate Matter Collected From Ambient Air," *Federal Register* 43 (194):46262-3.
- *Test Methods for Evaluating Solid Waste, Method 9022*, EPA Laboratory Manual, Vol. 1-A, SW-846.

2.3 Other Documents

- Crouthamel, C.E., et. al., *Applied Gamma Ray Spectrometry*, Pergamon Press, 1975.
- Lederer, C.M., et al., *Table of the Isotopes*, Sixth Edition, John Wiley and Sons, 1967.
- Shapiro, J., *Radiation Protection*, Harvard University Press, 1972.
- Faw, R., and Shultis, J., *Radiological Assessment*, Prentice - Hall, 1993.

- Heydorn, K., *Neutron Activation Analysis for Clinical Trace Element Research*, Vol. 1, CRC Press, 1984.
- Burn, R., *Research, Training, Test and Production Reactor Directory*, American Nuclear Society, Third Edition, 1988.

3. Summary of Method

3.1 NAA is an analytical technique dependent on the measurement of the number and energy of gamma and X-rays emitted by the radioactive isotopes produced in the sample matrix by irradiation with thermal neutrons from a nuclear reactor. Typically, the sample matrix plus appropriate blanks, SRMs, duplicates, and standards of the elements of interest are irradiated for a selected time period in the neutron flux core region of a research nuclear reactor. After irradiation and an appropriate radioactive decay, a gamma count-energy spectrum is obtained by counting the sample on a nuclear gamma spectroscopy detection system.

3.2 The NAA technique is qualitative in the sense that it incorporates the detection of characteristic radioisotopes (the neutron-induced radioactivity of each trace element emits a characteristic gamma ray energy spectrum, hence individual nuclear fingerprint); it is quantitative in that the detection system records not only the energy of a given gamma emission, but also the number of emissions per unit time.

3.3 Quantitative analysis is obtained by comparing the number of characteristic X-ray or gamma-rays of the unknown with the number determined for a standard. Interferences with a few elements must be corrected for using the intensity of related interference gamma peaks and computer programs for processing peak subtraction. Likewise, computer programs should also correct for sample count time, radioactive decay, sample weight or volume, reactor flux, and detector geometry. Calibration and efficiency of the detection system must be monitored throughout the counting period. Method detection limits (MDLs) for elements detected by NAA are listed in Table 2.

3.4 Method detection limits are intended as a guide to instrumental limits of a system optimized for multi-element determinations and employing commercial instrumentation and computer programs. However, actual MDLs are dependent on the sample matrix, instrumentations, and selected operating conditions of the particular nuclear research reactor.

4. Significance

4.1 In general, NAA is a versatile and non-labor intensive analytical technique. Liquids, solids, and gases can be analyzed by NAA. It is a multi-element technique in that many elements can be analyzed simultaneously in a given sample spectrum without changing or altering the apparatus as is necessary in atomic absorption, etc. Importantly, special sample preparations, such as digestion or extractions, are not required, and therefore the NAA method is non-destructive, i.e., the integrity of the sample is not changed in any manner by prechemistry or the addition of any foreign materials for irradiation. Thus, the problem of reagent-introduced contaminants is completely avoided. For most elements of interest, the analytical approach for NAA requires little if any post-irradiation chemistry; thus, the amount of technician contact per sample analysis is reduced considerably. The NAA method is fast and convenient; for many elements, several samples can be irradiated at a given time and counted later on a given decay schedule.

4.2 Finally, the NAA method offers high sensitivity to trace elements. The sensitivity obtained by activation analysis is a function of the neutron cross section of the element in question, available neutron flux, length of irradiation, resolution of the detector, matrix composition, and the total sample size. Hence, increasing neutron fluxes, increased irradiation times, and major advances in nuclear technology in the areas of increased efficiency and resolution have pushed the detection limits of most elements to very low levels.

5. Interferences

5.1 Elemental Nuclear Parameters. The radioactive elements, their related isotopes, and their nuclear parameters are shown in Table 3.

5.2 Interferences. Interferences with NAA occur in three primary categories as described in the following paragraphs.

5.2.1 Sensitivity. Sensitivity can decrease when an element exists in sufficient quantity to produce high gamma activity in the filter sample that masks the lower gamma activity from other elements of interest. The primary correction for this situation (when feasible) is to pick irradiation times and decay times that emphasize the elements of interest and de-emphasize the interfering element.

5.2.2 Similar gamma ray emissions. This situation exists when two radionuclides emit a gamma ray of similar energy that interferes with each other. Possible corrections to this situation are as follows:

- Utilize different irradiation and decay times.
- Use high-quality, high-resolution HPGe or GeLi detectors to resolve close peak, especially when using sophisticated computer programs that use iterative least square curve fitting and peak search routines.
- Use computer programs that locate a "clean" gamma photopeak of the interference and use its net area and known nuclear characteristics to calculate and subtract the interfering peak net area from the gamma ray of interest along with percent error calculations.

5.2.3 Duplicate radionuclide production. This interference occurs when the nuclei of 2 unrelated elements absorb a neutron and produce the exact same radionuclide. The correction is to use the net area of a gamma peak of another radionuclide associated with the interference and the known nuclear characteristics produced by the interference parent to calculate the actual production rates of the interference and subtract from the radionuclide gamma peak of interest along with percent error calculations.

6. Safety

6.1 Chemical Toxicology.

6.1.1 The toxicology or carcinogenicity of all chemicals used in this method is not completely known. Each chemical should be regarded as a potential health hazard, and exposure to these compounds should be as low as reasonably possible. Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

6.1.2 A reference file of material data handling sheets (MDS) also should be available to all personnel involved in the chemical analysis. All chemicals should have the purchase date and expiration date written on the container. Each professional or technician working in the laboratory should participate in yearly chemical safety refresher courses.

6.2 Radioactive Safety

6.2.1 The radiological operations should be under the direction of a radiological safety officer or health physicist.

6.2.2 The laboratory must have an Agreement State or NRC license to possess controlled levels of radioactivity.

6.2.3 All professionals and lab technicians should be officially trained, certified, and documented to work with radioactive materials.

6.2.4 Radiation surveys of the lab area work stations should be performed daily to properly limit the dose exposure to lab personnel and to lower the background levels of radioactivity subject to the nuclear detector systems used for NAA.

6.2.5 Contamination swipes of work areas should be performed weekly to protect employees from internal uptake and to prevent contamination of the nuclear detectors or specific samples being counted.

7. Definitions

[Note: Definitions used in this document are consistent with ASTM methods. All pertinent abbreviations and symbols are defined within this document at point of use.]

7.1 Instrument Detection Limit (IDL). The concentration equivalent of the element, which is equal to three times the standard deviation of the blank.

7.2 Method Detection Limit (MDL). The minimum concentration of the element that can be identified, measured, and reported with a 99% confidence level that the element concentration is greater than zero.

7.3 Laboratory Reagent Blank (LRB) (Preparation Blank). An aliquot of water that is treated exactly as a sample, including exposure to all labware, equipment, solvents, reagents, etc. that are used with other samples. The LRB is used to determine if the method analytes or other interferences are present in the laboratory environment, the reagents, or apparatus.

7.4 Internal Standard. Pure elements added to a solution in known amounts and used to measure the relative responses of other elements that are components of the same solution. The internal standard must be an element that is not a sample component.

7.5 Stock Standards Solution. A concentrated solution containing one or more elements prepared in the laboratory using certified reference compounds or purchased from a reputable commercial source.

7.6 Calibration Check. A radioactive standard (i.e., Ra²²⁶) used to determine acceptable instrument performance prior to calibration and sample analyses.

7.7 Laboratory Fortified Blank (LFB). An aliquot of reagent water to which known quantities of the method elements are added in the laboratory. The LFB is analyzed exactly like the sample, and its purpose is to determine whether method performance is within acceptable control limits.

7.8 Laboratory Fortified Sample Matrix (LFM). An aliquot of an environmental sample to which known quantities of the method elements are added in the laboratory. The LFB is analyzed exactly like the sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the elements in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for the concentrations found.

7.9 Quality Control Standard Reference Material (SRM's). Elementally certified standard reference materials (NIST, NRC, IAEA, etc.) of a matrix similar to the unknowns that are analyzed as blind control samples with the knowns.

8. Apparatus and Equipment

The basic instrumentation for performing neutron activation analysis consists of a nuclear reactor for irradiating the samples, nuclear detectors for detecting the gamma emissions, and various types of multi-channel analyzer systems that range from simple data acquisition systems to complex computerized data acquisition and processing systems. A general summary of the basic NAA process is shown in Figure 1.

8.1 Research Nuclear Reactor

8.1.1 The research nuclear reactor must have a minimum thermal neutron flux: 1×10^{13} n/cm²-s, open pool water cooled type containment, rotating, out-of-core, wet sample exposure facilities for uniform sample irradiation for 1 to 24 h, and pneumatic irradiation facilities for fast 10 s to 60 min sample irradiations.

8.1.2 Primary consideration must be given to accurately measuring the thermal neutron flux distribution within the volume that is intended for irradiation of the samples. Many conditions cause a distortion of the flux; hence, irradiation facilities need to be found where the flux does not vary over a few percent (2% or less). If this situation does not exist, small flux monitors must be located in the different sample positions for monitoring and counting the flux and making appropriate tables of flux correction factors.

8.2 Gamma Spectroscopy System

8.2.1 Large Volume Lithium-Drifted Coaxial Ge(Li) or HPGe Nuclear Detectors. The primary component in a gamma spectroscopy system is the gamma detector. This detector typically consists of a coaxial-shaped lithium-drifted germanium Ge(Li) crystal or a high-purity HPGe coaxial detector mounted in a vacuum tight cryostat, a liquid-nitrogen Dewar, and a very low noise preamplifier, all in one complete unit. These detectors are very sensitive to a wide energy range of gammas, from 0-3.0 MeV. Typical efficiencies for commercial detectors run from 10-50% with some possessing up to 100% efficiency. These detectors typically have an energy resolution of 1.7-2.2 keV and are capable of effectively providing a clear definitive response to the many different energy gammas in a typical irradiated sample. Due to their sensitivity to gamma rays from natural background radioactivity, Ge(Li) and HPGe detectors must be shielded by several inches of lead with a copper - cadmium inner liner. The nuclear characteristics of all radionuclides detectable on detectors such as these are shown in Table 4.

8.2.2 Low Energy Photon Detector (LEPD). A supplementary component of the gamma spectrometry system is a low energy photon detector (LEPD or LEPS). This detector is sensitive to low-energy photons (γ -rays and X-rays) and resolves X-ray events that are close in energy value. Since many elements, when bombarded with thermal neutrons in a reactor, give off X-rays and low energy γ -rays, the LEPD offers increased NAA sensitivity to many trace metals. The use of X-ray spectrometry when combined with a low energy photon detector has several notable advantages over routine γ -ray spectrometry. The isotopes decay with characteristic X-rays unique to the element; gamma decays are not necessarily so. Therefore, in an X-ray spectrum of a given matrix, many elements can be determined without the overlapping of photopeaks that sometimes occurs in γ -ray spectrometry. Another useful characteristic of the LEPD is that it is "blind" to the higher energy γ -rays emitted by the radioactive matrix. That is, the higher-energy photons tend to pass through the detector without interaction, while the low-energy particles are absorbed. The low-energy photon detector plus Dewar is very similar in size and shape to the standard large-volume Ge(Li) detectors. The LEPD unit is constructed of a virtually windowless (less than 1 μ m Ge) lithium-drifted germanium crystal wafer and is maintained at liquid nitrogen temperatures by a cryosorption pumping and a liquid nitrogen Dewar. The detector has a standard end cap window of 5 mils of beryllium. Such a detector, when coupled

with a 4096 or greater multichannel analyzer, has a useful range from 3 to approximately 600 keV. At above 500-600 keV, the relative photopeak efficiency drops off, but the resolution remains sufficient to separate photopeaks as close as 3 keV. At 270 keV, photopeaks as close as 750 keV can be separated. Typical resolution at 5.9 keV is 225 eV, and at 122 keV, it is 600 eV. The nuclear parameters of elements detected by LEPD detectors are illustrated in Table 5.

8.2.3 Data Acquisition Systems. The commercial multichannel analyzer (MCA) is a device whose primary function is to sort and store the myriad of proportion signals coming from the nuclear detector due to gamma interaction with the detector. Based upon the calibration of the system (usually 0 - 2 MeV for Ge(Li) and 0 - 200 keV for the LEPD), the analyzer will sort incoming signals according to their energy and store them in an appropriate memory bit or "channel." Such a system typically might have 4096 or 8192 channels and be attached to microprocessors that have simple data reduction routines for determining net X-rays counts in a peak and the X-rays energy of the peak. The more complex MCA data acquisition systems consist of either: (1) multiple 4096 channel analyzers connected to PC type computers with sufficient storage to have a variety of peak search and radionuclide identification routines, a library of reference standards, half-life decay corrections, etc.; the final product is a report listing the various experimental parameters of the sample and a listing of $\mu\text{g/g}$, etc. of each element present in the sample plus the error limits; or (2) multi-job, multi-task data acquisition processing systems, which allow flexibility in designing specific data acquisition areas for independently operating many separate nuclear detector systems with built-in microprocessors and minicomputers. The final product is the same as that for the PC type gamma spectroscopy systems.

9. Reagents and Consumable Materials

9.1 Reagents

[Note: For purposes such as cleaning the hi-purity irradiation vials, acidifying solutions for storage, and conducting standard dilution procedures, etc., sources of high-purity nitric acid should be maintained.]

9.1.1 Nitric Acid, high-purity, concentrated (sp.gr.1.41). Commercial Sources.

9.1.2 Water. ASTM Type I water (ASTM D1193) for use in cleaning procedures, standard dilutions, etc.

9.2 Standard Stock Solutions

[Note: High-quality commercial grades of (atomic absorption) elemental standard solutions plus mixed element standards can be effectively used.]

9.2.1 For the elements Ti, I, Cu, Mg, Mn, Na, K, Cl, V, and Al, the acid base for the solution must be nitric acid. Both HCl and H_2SO_4 produce interferences in the standards for short-lived elements that are not acceptable.

9.2.2 For all other elements, either a HNO_3 or HCl acid base is suitable.

9.2.3 Both single and multi-element standards should be prepared in bulk in the different size irradiation container volumes used to eliminate stability problems.

9.3 Blanks

A laboratory reagent blank (LRB) is used to determine potential contamination problems and to determine background levels of the elements of interest. The LRB must contain all the reagents in the same volumes as used in preparing the samples. It must be carried through the sample preparation scheme.

9.4 Quality Control (QC) Standard Reference Materials

The SRMs must be obtained from commercial (outside the lab) and federal sources of certified standard reference materials. Certification documents and expiration dates must be maintained.

9.5 Sample Irradiation Vials

Pre-cleaned, high-purity Kartell or Olympic linear polyethylene vials in the 1 mL, 2.5 mL, 5 mL, 10 mL, and 20 mL size should be maintained. A device needs to be maintained in a clean hood for heat-sealing all sample vials.

10. Sample Receipt in the Laboratory

As you are notified of their availability, personnel within the neutron activation analysis laboratory shall receive the samples as they are mailed or hand carried. Upon receipt of samples, assign each job a control number and the chain of custody documents. Assign all samples an ascending NAA Lab number in the NAA Laboratory Sample Receipt Log. Store all samples requiring refrigeration in refrigerator-freezers. All other samples should be stored under cool, dry, and dark conditions in laboratory storage cabinets in the restricted areas of the laboratory. (Samples requiring desiccant storage are stored in that manner). Limit all contact with these samples to only the personnel assigned to this project.

11. Calibration

Ge(Li) or HPGe gamma spectroscopy detection systems are generally operated on an energy range of 0 to 2000 keV utilizing 4096 to 8192 channels of data acquisition storage. The three calibration items of daily interest are energy calibration, detector peak resolution, and efficiency stability.

11.1 Maintain a radioactive source (i.e. Ra-226 or an NIST multi-radionuclide source) that provides gamma emissions from 50-1,750 keV.

11.2 Maintain this calibration source in a condition that allows a fixed, unchanged geometry that can be duplicated exactly each time it is used.

11.3 Arrange for computer programs that allow daily comparisons of an initial first count of this standard to all future daily counts.

11.4 Set up a daily QA/QC procedure for each detector that has the following:

- Duplicated geometry.
- Duplicated count time.
- Duplicated computer program parameters.

- Duplicated count comparisons that are compared to initial settings.

11.5 Count the calibration standard and process data.

11.6 For the energy calibration check, review all the designated gamma peak measurements to determine that all peaks are within ± 0.5 keV of their true value.

11.7 For the peak resolution check, review all peaks for anomalies, but concentrate on the 1,172-1,333 keV region to insure a peak resolution of 2.2 keV or less.

11.8 For the efficiency resolution check, compare peaks in the 500 to 600 keV and the 1100 to 1300 keV energy range to determine that the net gamma count in those peaks (corrected for decay) have not varied more than $\pm 5.0\%$ from the initial preset measured values.

12. Quality Control (QC)

For neutron activation analysis methods, it is recommended that a documented, certified QA/QC program utilizing validated SOPs and QA/QC document forms be established. A series of standard operating procedures should be defined in the following areas for quality control of the complete analytical procedure. Documented performance records showing quality of performance should be maintained.

12.1 When samples arrive, verify them against shipping documents. Notify discrepancies and assign control numbers. Record the samples in the entry log, and assign appropriate (matrix related) storage. An analytical job plan should also be formatted. The following control forms and concepts should be developed and used:

- Sample Receipt Control Numbers.
- Chain of Custody Document Form.
- Unique Sample I.D. Numbers.
- Daily Freezer and Refrigerator Temperature Checks recorded on forms.
- Controlled Access Areas.

12.2 Maintain all laboratory areas in a controlled, clean, and organized atmosphere. The following control forms should be developed and used:

- Daily Hood System Flow Checks.
- Daily Radiation Surveys of the Lab Work Area recorded on forms.
- Weekly Laboratory Radiation Swipe Tests recorded on forms.

12.3 All employees of the laboratory shall have documented training programs. The following control forms should be developed and used:

- Employee Systems Operation Certifications (annual).
- Employee Update Training (annual).
- Employee SOP Certification.

12.4 Prepare all analytical standards and unknowns (as required) in class 100 clean hood conditions according to documented instructions. The following control forms should be developed and used:

- Unknown Preparation Form.
- Standard Preparation Form.
- SRM, Blank, and Duplicates Preparation Form.

- Shelf-Life Certifications.
- Sample Weight QA form.
- Sample Volume QA form.

12.5 Prior to use each day, check and certify each weight balance system (using NIST certified weights). Each technician using the systems certifies balance compliance. Records are stored on all certifications. A Daily Balance Calibration Checks control form should be developed for this activity.

12.6 Routinely check pipettes for volume certification using the weighing of deionized water of known volumes. Records are stored on all certifications. A Weekly Pipette Calibration Checks control form should be developed for this activity.

12.7 Conduct a daily calibration, efficiency, and resolution check (using an NIST source, etc.) on each gamma spectroscopy system. A (sign-off) daily review of certification should be made prior to use by each technician using the system. Stored records are maintained on all QA data. The following control forms and concepts should be developed and used:

- Daily QA/QC Calibration, Efficiency, and Resolution Checks.
- Daily QA/QC Performance Charts.
- Weekly QA/QC Background Checks.
- System Maintenance Logs.

12.8 Nuclear reactor flux monitor irradiations will be made and accurate flux monitor corrections maintained for the irradiation locations in each of the wet exposure irradiation ports. Certified records should be maintained for all data and calculations related to these flux correction factors. These documents are:

- NIST Flux Correction Tables.
- Flux Raw Data Documents and Experimental Parameters.

12.9 For each nuclear detector used in the system, determine all geometry (different sample counting configurations) correction factors (curves). The following documents should be maintained:

- Certified Geometry Correction Factors Tables.
- Geometry Raw Data Documents and Experiment Parameters.

12.10 Prepare validation documents for the following:

- SOPs.
- All Computer Programs Used.

12.11 Maintain computer backup disks at a different location for the following:

- Gamma Spectroscopy Data Acquisition and Data Processing Programs.
- System QA/QC Gamma Spectroscopy Data.
- Client Project Gamma Spectra and Nuclear Parameters for Samples, Standards, Duplicates, SRMs and Blanks.

12.12 Thoroughly document all sample irradiation and counting. The following control forms should be developed and used:

- Nuclear Reactor Run Sheet.
- Pneumatic Facility Operation Form.
- Sample Counting and Data Processing Form.

12.13 For QA/QC control, a batch of NAA samples is considered to be a group of samples irradiated at the same time under the same irradiation conditions, counted on the same gamma spectroscopy detectors and data acquisition systems, and processed under the exact same count time, calibration, efficiency, and geometry conditions. Each NAA sample batch should contain the following QC samples as a minimum:

- Blank Vial Analyses - 1 blank vial per 20 unknown samples.
- NBS SRM Analyses - 2 SRMs per 20 unknown samples.
- Duplicate Unknown Analyses - 1 duplicate per 20 unknown samples.
- Standards - Triplicate standards per batch of unknowns.

12.14 Use data review forms to evaluate the performance of these categories for each batch (and monthly summaries) with the following controls:

- Duplicates: Solids = $\pm 20\%$ variance.
Liquids = $\pm 10\%$ variance.
Air = $\pm 20\%$ variance.
- Method blanks: $< 3X$ method blank average.
- SRMs: Solids $\pm 10\%$ certified value.
Liquids $\pm 5\%$ certified value.
Air = $\pm 20\%$ certified value.
- Standards: $\pm 1\%$ variance.

12.15 The following system manuals and training documents should be maintained:

- Gamma Spectroscopy System Manuals.
- Computer Program Software Manuals.
- Radiation Safety Study Guide (Health Physics Manual).
- QA/QC Validation Guidelines.
- QA/QC Lab Inspections.
- *Applied Gamma - Ray Spectrometry*, Adams and Dams.
- *Table of the Isotopes*, Lederer, Hollander & Perlman.
- SOP Manual
- Detector Geometry Correction Manual
- Reactor Flux Correction Manual
- Material Safety Data Sheet Manual

13. Procedure

13.1 Unknown Sample Preparation

13.1.1 Pre-punch air filter samples to a 3.0 cm diameter. All sample handling should be performed in laminar flow clean hoods using new disposable plastic gloves, etc.

13.1.2 Carefully fold each air filter disk into itself until it is 1/4 its original size. See Figure 2 for an illustration of the folding process.

13.1.3 Carefully insert the filter into pre-cleaned Kartell or Olympic hi-purity linear polyethylene irradiation vials that are 30 mm long and 8 mm in diameter. Put the sample I.D. number on the vial using marking pens that use ink that contains none of the elements of interest. Heat-seal and pre-clean the vial prior to irradiation. All sample preparation should be controlled by instructions on the unknown QA/QC form.

Each of the new Kartell or Olympic hi-purity irradiation polyethylene vials are treated as follows prior to placing samples into them for irradiation:

- 10 min soak in 20% nitric acid-distilled water solution.
- 60 min soak in deionized water.
- Rinse in distilled water.

In each batch of new irradiation vials, a total of 10 of each type vial and trace element levels determined to ensure that contamination of the samples by the vial will not occur.

13.2 Metal Blank Preparation

13.2.1 Prepare method blank filters using the same procedures identified in Section 13.1 for unknowns.

13.2.2 Prepare a minimum of one method blank filter per 20 unknown samples.

13.3 Duplicate Sample Preparation

13.3.1 Prepare duplicate sample filters using the same procedures identified in Section 13.1 for unknown filter being duplicated.

13.3.2 Prepare a minimum of one duplicate filter per 20 unknown samples.

13.4 Standards Preparation

13.4.1 Prepare or purchase mixed element standards, liquid or solid, including the three categories of standards itemized in Table 6.

13.4.2 Using the instructions on the QA/QC Standards preparation form, carefully pipette (using QA/QC certified and checked calibrated pipettes) 0.5 ml of each mixed standard solution into its own unique (pre-cleaned) Kartel or Olympic plastic 30 mm by 8 mm irradiation vial that has been labeled as follows:

SMES (Short Mixed Element Standard)

MMES (Medium Mixed Element Standard)

LMES (Long Mixed Element Standard)

Next trim, heat-seal, and clean vials as per the instructions for the unknowns.

13.4.3 Standards should be prepared in triplicate based on the elements that are being analyzed in the air particulate filters.

13.5 Irradiation and Counting for Short - Lived Isotopes

Unknown samples, standards, blanks, duplicates, and SRMs are irradiated individually for 20 s to 10 min in the pneumatic transfer system of the nuclear reactor at a flux of 1.0×10^{13} n/cm² -sec. (or greater). After a monitored decay, each sample vial is rinsed in a radioactive hood and placed in non-irradiated counting vials. After an exact decay time (determined by the results of preliminary sample analyses), each sample, standard, blank, duplicate and SRM is counted for 30 to 300 s on designated Ge(Li) detection systems. Data is analyzed (using the nuclear parameters in Tables 2 and 3) and spectra are permanently stored on disk with appropriate nuclear parameters included. The elements typically analyzed by short-lived isotopes are Al, V, Ti, Mn, Mg, Cl, Cu, F, O, Na, K, I, etc.

13.6 Irradiations and Counting for Medium and Long - Lived Isotopes

Unknown samples, standards, blanks, duplicates, and SRMs are again irradiated for 1 to 24 h at a flux of 1.0×10^{13} n/cm²-s. (or greater) in the rotating wet vertical exposure irradiation facilities of the nuclear reactor. After approximately a 5-7 day radioactive decay (to allow interfering activities to decay away) of the samples and reference standards, both unknowns, SRMs, and standards are counted for 400-1,200 s on the gamma detection systems. Data is analyzed (using the nuclear reactor parameters in Tables 4 and 5) and spectra is permanently stored on disk with appropriate decay time, dead time, etc. and other nuclear data included. The elements typically analyzed by medium-lived isotopes are Na, K, As, Sm, Cd, La, Mo, Br, Sb, U, Hg, Au, W, Pt, etc. After a 14-21 day decay, a 1,000-1,800 s gamma count of each sample and standard is made for long-lived isotopes. The elements typically analyzed by long-lived isotopes are Ce, Ca, Lu, Th, Cr, Eu, Yb, Nd, Zr, Ag, Cs, Fe, Sc, Zn, Co, Sr, Rb, Ni, Ba, etc. (using nuclear parameters shown in Tables 4 and 5).

14. Calculations and Data Processing

Spectral data from the unknown samples, standards, and SRMs are processed using the commercially developed computer programs for gamma peak search, least squares fitting, and NAA quantitative analysis. All spectra and associated nuclear parameters from the analyses are stored on disk for future access as needed. Results are reported as µgrams or nanograms element per cubic meter of air with percent error limits (2 sigma) based on known errors associated with counting statistics, precision, accuracy of standards, weighing, pipetting, etc. As part of the computer processing during the counting phase, an automatic peak search performs a gaussian peak fit on all single peaks. Multiples (several peaks close together) are analyzed by an iterative least squares gaussian fit. The result from this peak program is the determination of the centroid channel (energy), FWHM (full width-half max), net integrated peak area, and background. When no peak area appears, the system calculates a less-than value (based on all available nuclear parameters) for the elemental determination. The number of counts per sec per cubic meter of air, etc. of sample (N_a) is computed as:

$$N_a = \frac{N}{(V_{std})(t_c)(D)(F)(G)}$$

where:

- N = the number of counts observed (net peak area).
- V_{std} = the standard cubic meters of air sampled (m³)
- F = the sample reactor flux position correction.
- G = the sample geometry correction.
- t_c = the live counting time in seconds established for each count.
- D = a dimensionless decay correction related to the end of the sample irradiation and is computed by:

$$D = \exp. \left[\frac{(0.693t)}{(T_{1/2})} \right]$$

where:

- t = the elapsed time from end of irradiation to the middle of the counting sequence in the same units as T.
- T = the half-life of the element being evaluated.

The number of μg , etc. of element per cubic meter of air is then calculated by dividing (N_a) by (N_s), which is the analogous term to (N_a) but obtained for a standard. The output results for each element are represented in units of $\mu\text{g}/\text{m}^3$. A sample data output report is provided as Figure 3.

15. Precision and Accuracy

Precision is measured as the percent difference between the values obtained for duplicates (i.e., N_{a1} and N_{a2}). The average of the values is used to base the percentage difference. The measurement of precision provides us with the best indication of quality control's success. This determination is shown in the sum of errors associated with weighing, pipetting, individual reactor irradiations, counting statistics, detector efficiency and resolution, and sample homogeneity.

$$\% D = \left[\frac{ \left(\frac{N_{a2} + N_{a1}}{2} \right) - \left(\frac{N_{a1} + N_{a2}}{2} \right) }{ \left(\frac{N_{a1} + N_{a2}}{2} \right) } \right] \times 100$$

15.1 Precision errors associated with typical reactor flux changes in pool type research reactors are illustrated in Table 7.

15.2 Typical precision errors associated with detector efficiency changes with time are illustrated in Table 8.

15.3 Typical precision errors associated with all variables involved in the preparation of samples are illustrated in Table 9.

15.4 Accuracy

Accuracy is measured by percent error for standards and the results for analysis of various Standard Reference Materials from NIST, Canada, IAEA, and other related agencies. Percent error is expressed as the following equation:

$$\% \text{ Error} = \left[\frac{(\text{Observed} \ \& \ \text{Known})}{(\text{Known})} \right] \times 100$$

15.4.1 Tables 10 through 13 represent a typical accuracy check in various matrix Standard Reference Materials that are routinely irradiated and analyzed with unknowns.

15.4.2 For accurate QA control, a wide variety of SRMs should be maintained in the laboratory similar to the typical ones listed in Table 14.

16. References

1. Crouthamel, C.E., et al., *Applied Gamma Ray Spectrometry*, Pergamon Press, 1975.
2. Lederer, C.M., et al., *Table of the Isotopes*, 6th Edition, John Wiley and Sons, 1967.
3. U. S. Environmental Protection Agency, *Test Method for Evaluating Solid Waste*, Method 9022, EPA Laboratory Manual, Vol. #1 - A, SW-846.
4. Shapiro, J., *Radiation Protection*, Harvard University Press, 1972.
5. Faw, R., and Shultis, J. *Radiological Assessment*, Prentice - Hall, 1993.
6. Heydorn, K., *Neutron Activation Analysis for Clinical Trace Element Research*, CRC Press, Vol. 1, 1984.
7. Burn, R., *Research, Training, Test and Production Reactor Directory*, American Nuclear Society, Third Edition, 1988.

TABLE 1. EXAMPLE SUMMARY OF ELEMENTS COMPATIBLE WITH NAA

ELEMENT	SYMBOL	Chemical Abstract Services Registry Numbers (CASRN)
TITANIUM	Ti	7440 - 32 - 6
TIN	Sn	7440 - 31 - 5
IODINE	I	7553 - 56 - 2
MANGANESE	Mn	7439 - 96 - 5
MAGNESIUM	Mg	7439 - 95 - 4
COPPER	Cu	7440 - 50 - 8
VANADIUM	V	7440 - 62 - 2
CHLORINE	Cl	7782 - 50 - 5
ALUMINUM	Al	7429 - 90 - 5
MERCURY	Hg	7439 - 97 - 6
SAMARIUM	Sm	7440 - 19 - 9
TUNGSTEN	W	7440 - 33 - 7
MOLYBDENUM	Mo	7439 - 98 - 7
URANIUM	U	7440 - 61 - 1
LANTHANUM	La	7439 - 91 - 0
CADMIUM	Cd	7440 - 43 - 9
ARSENIC	As	7440 - 38 - 2
ANTIMONY	Sb	7440 - 36 - 0
BROMINE	Br	7726 - 95 - 6
SODIUM	Na	7440 - 23 - 5
POTASSIUM	K	7440 - 09 - 7
CERIUM	Ce	7440 - 45 - 1
CALCIUM	Ca	7440 - 70 - 2
LUTETIUM	Lu	7439 - 94 - 3
SELENIUM	Se	7782 - 49 - 2
TERBIUM	Tb	7440 - 27 - 9
THORIUM	Th	7440 - 29 - 1
CHROMIUM	Cr	7440 - 47 - 3
EUROPIUM	Eu	7440 - 53 - 1
YTTERBIUM	Yb	7440 - 64 - 4
HAFNIUM	Hf	7440 - 58 - 6
BARIUM	Ba	7440 - 39 - 3
STRONTIUM	Sr	7440 - 24 - 6
NEODYMIUM	Nd	7440 - 00 - 8
SILVER	Ag	7440 - 22 - 4
ZIRCONIUM	Zr	7440 - 67 - 7
CESIUM	Cs	7440 - 46 - 2
NICKEL	Ni	7440 - 02 - 0
SCANDIUM	Sc	7440 - 20 - 2
RUBIDIUM	Rb	7440 - 17 - 7
IRON	Fe	7439 - 89 - 6
ZINC	Zn	7440 - 66 - 6
COBALT	Co	7440 - 48 - 4
TANTALUM	Ta	7440 - 25 - 7

TABLE 2. EXAMPLE OF ESTIMATED NEUTRON ACTIVATION ANALYSIS (NAA) METHOD
DETECTION LIMITS (MDLs) IN NANOGRAMS¹ AND ng/m³²

Element	Method Detection Limits (MDLs)		Element	Method Detection Limits (MDLs)		Element	Method Detection Limits (MDLs)	
	ng	ng/m ³		ng	ng/m ³		ng	ng/m ³
ALUMINUM	1	5.0	INDIUM	0.005	0.02	SAMARIUM	0.009	0.04
ANTIMONY	0.09	0.4	IODINE	1	5.0	SCANDIUM	0.200	0.09
ARGON	0.01	0.04	IRIDIUM	0.002	9.2x10 ⁻³	SELENIUM	2.00	9.2
ARSENIC	0.02	0.09	IRON	1000	4629.0	SILVER	0.01	0.04
BARIUM	0.5	2.3	KRYPTON	0.1	0.5	SODIUM	0.04	0.18
BROMINE	0.01	0.04	LANTHANUM	0.04	0.2	STRONTIUM	0.09	0.40
CADMIUM	0.9	4.2	LUTETIUM	0.0009	4.2x10 ⁻³	TANTALUM	0.90	4.2
CALCIUM	50	231.0	MAGNESIUM	50	231.0	TELLURIUM	1.00	4.6
CERIUM	2	9.2	MANGANESE	0.005	0.02	TERBIUM	0.500	2.3
CESIUM	0.04	0.02	MERCURY	0.2	0.9	THORIUM	0.700	3.2
CHLORINE	2	9.2	MOLYBDENUM	1	5.0	TIN	2.00	9.2
CHROMIUM	20.0	92.5	NEODYMIUM	2	9.2	TITANIUM	1.00	4.6
COBALT	0.09	0.4	NICKEL	0.4	1.8	TUNGSTEN	0.02	0.09
COPPER	0.20	0.9	NIObIUM	10	46.0	URANIUM	0.04	0.20
DYSPROSIUM	0.000004	1x10 ⁻⁵	PALLADIUM	0.02	0.09	VANADIUM	0.01	0.04
ERBIUM	0.04	0.2	PLATINUM	0.4	1.8	YTTERBIUM	0.04	0.20
EUROPIUM	0.00009	4.1x10 ⁻⁴	POTASSIUM	0.2	0.92	YTRIUM	0.02	0.09
FLUORINE	20.0	92.5	PRASEODYMIUM	0.01	0.04	ZINC	2.00	9.2
GADOLINIUM	2.00	9.2	RHENIUM	0.004	0.02	ZIRCONIUM	20.00	92.6
GOLD	0.02	0.09	RHODIUM	0.002	9.2x10 ⁻³			
HAFNIUM	0.70	8.2	RUBIDIUM	4.00	18.5			
HOLMIUM	0.00	0.02	RUTHENIUM	0.200	0.90			

¹Results listed are based on a 10 h irradiation at 1 x 10¹³ n/cm-s with no interfering elements or matrix effects.

²Based upon dichotomous sampling for 24-hrs using a 37-min Teflon® filter at a sampling rate of 0.9 m³/hr.

TABLE 3. GAMMA RAY INTERFERENCES

Element	Isotope	Half-Life	Energy (keV)	Interfering Isotope	Half-Life	Interference (keV)	Reference (keV)	Abundance Ratio
Dysprosium	¹⁶⁵ Dy	2.33 h	94.7	²³³ Th	22.3 m	94.7	86.5	0.346
Magnesium	²⁷ Mg	9.46 m	843.8	⁵⁶ Mn	2.58 h	846.8	1810.7	3.636
Manganese	⁵⁶ Mn	2.58 h	846.8	²⁷ Mg	9.46 m	843.8	1014.4	2.496
Antimony	¹²² Sb	2.70 d	564.2	⁷⁶ As ¹³⁴ Cs	26.32 h 2.06 y	563.2 563.2	559.1 795.8	0.0267 0.0981
Arsenic	⁷⁶ As	26.32 h	559.1	^{114m} In	49.51 d	558.4	190.3	0.0649
Bromine	⁸² Br ⁸² Br	35.3 h 35.3 h	619.1 776.5	¹⁸⁷ W ⁹⁹ Mo	23.9 h 65.94 h	618.3 778.0	685.7 739.6	0.2300 0.3578
Calcium	⁴⁷ Ca	4.54 d	1297.1	¹⁵² Eu	13.33 y	1299.1	1408.1	0.0784
Gallium	⁷² Ga	14.1 h	834.1	⁵⁴ Mn	312.12 d	834.8	-----	-----
Germanium	⁷⁷ Ge	11.3 h	264.4	⁷⁵ Se	119.77 d	264.7	400.7	5.121
Gold	¹⁹⁸ Au	2.70 d	411.8	¹⁵² Eu	13.33 y	411.1	344.3	0.0839
Holmium	¹⁶⁶ Ho	26.8 h	80.6	¹³¹ I	8.04 d	80.2	364.5	0.0323
Tungsten	¹⁸⁷ W ¹⁸⁷ W	23.9 h 23.9 h	134.2 685.7	¹³¹ Ba ¹⁸¹ Hf ^{110m} Ag	11.8 d 42.39 d 249.76 d	133.6 133.0 687.0	216.0 345.9 657.8	0.1095 2.3800 0.0681
Uranium	²³⁹ Np ²³⁹ Np	2.36 d 36 d	228.2 277.6	¹³² Te ¹⁸² Ta ²³² Th ¹⁴⁷ Nb ¹⁵² Eu	78.2 h 114.5 d 1.4E+ 10y 10.98 d 13.33 y	228.3 229.3 277.4 275.4 275.4	49.8 222.1 583.2 531.0 344.3	6.1250 0.4790 0.0747 0.0611 0.0013
Ytterbium	¹⁷⁵ Yb	4.19 d	282.5	^{177m} Lu	160.9 d	281.8	208.4	0.2315
Antimony	¹²⁴ Sb	60.2 d	602.7	¹³⁴ Cs	2.06 y	604.7	795.8	1.1418

TABLE 3. (continued)

Element	Isotope	Half-Life	Energy (keV)	Interfering Isotope	Half-Life	Interference (keV)	Reference (keV)	Abundance Ratio
Cerium	¹⁴¹ Ce	32.5 d	145.4	¹⁸³ Ta ¹⁷⁵ Yb	5.10 d 4.19 d	144.1	246.1	0.0954
						144.9	396.3	0.0511
Cesium	¹³⁴ Cs ¹³⁴ Cs	2.06 y 2.06 y	604.7 795.8	¹²⁴ Sb ²³² Th	60.2 d 1.4E+10y	602.7 795.0	1691.0 911.2	2.0660 0.1632
Chromium	⁵¹ Cr	27.7 d	32.1	¹⁴⁷ Nb ^{177m} Lu	10.98 d 160.9 d	319.4 319.0	531.0 378.5	0.1488 0.3971
Hafnium	¹⁸¹ Hf ¹⁸¹ Hf	42.39 d 42.39 d	133.0 482.2	¹³¹ Ba ¹¹⁴ Ce	11.8 d 284.9 d	133.6 133.5	216.0 -----	0.1095 -----
Indium	^{114m} In	49.51 d 49.51 d	190.3	⁵⁹ Fe	44.5 d 44.5 d	192.4 192.4	1099.2 1099.2	0.0545 0.0545
Iridium	¹⁹² Ir ¹⁹² Ir	73.83 d 73.83 d	296.0 468.1	^{177m} Lu ²³⁸ U	160.9 d 4.5E+09y	296.4 295.2	378.5 351.9	0.1949 0.5175
Iron	⁵⁹ Fe	44.5 d 44.5 d	1291.6	^{115m} Cd ¹⁸² Ta	44.6 d 114.5 d	1290.6 1289.2	933.8 1221.4	0.4450 0.0461
Mercury	²⁰³ Hg	46.61 d 46.61 d	279.2	⁷⁵ Se	119.77d 119.77d	279.5 279.5	264.7 264.7	0.4257 0.4257
Nickel	⁵⁸ Co	70.82 d 70.82 d	810.8	¹⁵² Eu	13.33 y 13.33 y	810.8 810.8	778.9 778.9	0.0244 0.0244
Osmium	¹⁸⁵ Os	93.6 d 93.6 d	646.1	¹²⁴ Sb	60.2 d 60.2 d	645.9 645.9	602.7 602.7	0.0755 0.0755
Ruthenium	¹⁰³ Ru	32.26 d 32.26 d	497.1	¹³¹ Ba	11.8 y 11.8 y	496.3 496.3	373.2 373.2	3.308 3.308
Scandium	⁴⁶ Sc	83.81 d 83.81 d	1120.6	¹⁸² Ta ²³⁸ U	114.5 d 4.5E+09y	1121.3 1120.3	1221.4 609.3	1.2915 0.3254
Selenium	⁷⁵ Se	119.77 d 119.77 d	264.7	¹⁸² Ta	114.5 d 114.5 d	264.1 264.1	222.1 222.1	0.4789 0.4789
Silver	^{110m} Ag ^{110m} Ag	249.76 d 249.76 d	657.8 884.7	¹⁵² Eu ¹⁹² Ir	13.33 y 73.83 d	656.5 884.5	778.9 316.5	0.0110 0.0034

TABLE 3. (continued)

Element	Isotope	Half-Life	Energy (keV)	Interfering Isotope	Half-Life	Interference (keV)	Reference (keV)	Abundance Ratio
Thorium	²³³ Pa	27.0 d	300.2	¹⁶⁰ Tb ²³² Th	72.3 d 1.4E+10y	298.6 300.1	879.4 238.6	0.8781 0.0623
		27.0 d	312.0	⁴² K	12.36 h	312.4	1524.7	0.0186
Thulium	¹⁷⁰ Tm	128.6 d	84.3	¹⁸² Ta	14.5 d	84.7	100.1	0.1943
Tin	¹¹³ Sn	115.09 d	391.7	¹⁶⁰ Tb	72.3 d	392.5	298.6	0.0496
Ytterbium	¹⁶⁹ Yb	32.02 d	177.2	^{177m} Lu	160.9 d	177.0	378.5	0.1235
		32.02 d	198.0	¹⁶⁰ Tb ¹⁸² Ta	72.3 d 114.5 d	197.0 198.4	298.6 222.1	0.1940 0.1987
Zinc	⁶⁵ Zn	243.9 d	1115.6	⁴⁶ Sc ¹⁵² Eu ¹⁶⁰ Tb ¹⁸² Ta	83.81 d 13.33 y 72.3 d 114.5 d	1120.6 1112.1 1115.1 1113.4	889.3 1408.0 1178.0 1221.4	1.0000 0.6475 0.1000 0.0160
		64.02 d	724.2	¹²⁴ Sb ¹⁵⁴ Eu	60.2 d 8.59 y	722.8 723.3	602.7 1274.8	0.1115 0.5549
		64.02 d	756.7	¹⁵⁴ Eu	8.59 y	756.9	1274.8	0.1115
		34.97 y	765.8	¹⁵² Eu ¹⁶⁰ Tb	13.33 y 72.3 d	764.9 765.3	778.9 879.4	0.0139 0.0670

TABLE 4. NUCLEAR PARAMETERS FOR NEUTRON ACTIVATION ANALYSIS

Element	Stable isotope	Abundance (%)	Neutron activation cross section (barn)			1 / F	Radionuclide formed	Half-Life	Most useful (-ray)	
			Thermal (F)	Epithermal (I)					Energy (keV)	Abundance (%)
F	¹⁹ F	100	0.0098	0.04	4.1	²⁰ F	11.4 s	1634	100	
Na	²³ Na	100	0.528	0.31	0.59	²⁴ Na	15.0 hr	1368	100	
Mg	²⁶ Mg	11.2	0.0038	0.03	7.9	²⁷ Mg	9.5 min	844	70	
Al	²⁷ Al	100	0.232	0.17	0.73	²⁸ Al	2.3 min	1779	100	
S	³⁶ S	0.015	0.15	--	--	³⁷ S	5.1 min	3105	90	
Cl	³⁷ Cl	24.5	0.43	0.17	0.41	³⁸ Cl	37.3 min	2168	100	
K	⁴¹ K	6.8	1.48	1.28	0.86	⁴² K	12.4 hr	1525	18	
Ca	⁴⁶ Ca	0.0033	0.7	0.32	0.46	⁴⁷ Ca	4.6 d	1297	74	
--	--	--	--	--	--	⁴⁷ Sc	3.4 d	160	73	
--	⁴⁸ Ca	0.185	1.1	0.90	0.82	⁴⁹ Ca	8.8 min	3084	89	
Sc	⁴⁵ Sc	100	10	--	--	^{46m} Sc	20 s	143	46	
--	--	--	25	11	0.44	⁴⁶ Sc	84 d	889	100	
Ti	⁵⁰ Ti	5.3	0.179	~ 5	~ 30	⁵¹ Ti	5.8 min	320	95	
V	⁵¹ V	100	4.90	2.7	0.55	⁵² V	3.75 min	1434	100	
Cr	⁵⁰ Cr	4.3	16.0	8.5	0.53	⁵¹ Cr	27.8 d	320	9	
Mn	⁵⁵ Mn	100	13.3	14	1.1	⁵⁶ Mn	2.58 hr	847	99	
Fe	⁵⁸ Fe	0.31	1.14	1.2	1.1	⁵⁹ Fe	45 d	1099	56	
Co	⁵⁹ Co	100	19.9	--	--	^{60m} Co	10.4 min	59	2.1	
--	--	--	37.5	75	2	⁶⁰ Co	5.2 yr	1333	100	
Ni	⁵⁸ Ni	67.8	--	--	--	⁶⁵ Ni	71 d.	811	99	
--	⁶⁴ Ni	1.16	1.50	0.9	0.6	⁶⁵ Ni	2.55 hr	1482	25	

TABLE 4. (continued)

Stable isotope			Neutron activation cross section (barn)				Radionuclide formed			Most useful (-ray)	
Element	Stable isotope	Abundance (%)	Thermal (F)	Epithermal (I)	1 / F	Half-Life	Energy (keV)	Abundance (%)			
--	⁶⁸ Zn	18.6	0.07	0.24	3.4	13.9 hr	439	95			
Ga	⁷¹ Ga	39.5	4.7	25	0.53	14.1 hr	834	96			
Ge	⁷⁰ Ge	20.6	3.5	--	--	11.4 d	Ga x ray	--			
--	⁷⁴ Ge	36.7	0.52	0.9	1.7	83 min	265	11			
As	⁷⁵ As	100	4.4	63	14	26.4 hr	559	43			
Se	⁷⁶ Se	0.9	55	500	9.1	120 d	265	60			
--	⁷⁶ Se	9	21	42	2.0	17.4 s	162	50			
Br	⁷⁹ Br	50.5	2.6	--	--	4.4 hr	37	36			
--	--	--	11.0	125	11	17 min	616	7			
--	⁸¹ Br	49.5	3.0	50	17	35.3 hr	777	83			
Rb	⁸⁵ Rb	72.1	0.45	7	16	18.7 d	1079	8.8			
--	⁸⁷ Rb	27.9	0.12	2.3	19	17.7 min	1836	21			
Sr	⁸⁸ Sr	0.55	0.7	7.5	11	65 d	514	100			
--	⁸⁸ Sr	9.9	0.8	4	5	2.8 hr	389	80			
Y	⁸⁹ Y	100	0.0010	0.89	890	3.1 hr	202	97			
Zr	⁹⁰ Zr	17.4	0.0075	0.38	51	65 d	757	49			
--	--	--	--	--	--	35 d	766	100			
--	⁹¹ Zr	2.8	0.05	5.0	100	17.0 hr	743	99			
--	--	--	--	--	--	72 min	658	98			
Nb	⁹³ Nb	100	0.15	8.0	53	6.3 min	Nb x rays	--			
Mo	⁹⁸ Mo	23.8	0.14	7.5	54	67 hr	740	12			

TABLE 4. (continued)

Element			Neutron activation cross section (barn)				Radionuclide formed			Most useful (-ray)	
Element	Stable isotope	Abundance (%)	Thermal (F)	Epithermal (I)	1 / F	Radionuclide formed	Half-Life	Energy (keV)	Abundance (%)		
--	--	--	--	--	--	¹⁰¹ Tc	14.0 min	307	91		
Ru	⁹⁹ Ru	5.5	0.21	5.5	26	⁹⁷ Ru	2.8 d	215	91		
--	¹⁰² Ru	31.6	1.3	4.2	3.2	¹⁰³ Ru	39.5 d	497	88		
--	¹⁰⁴ Ru	18.8	0.47	5.2	11	¹⁰⁵ Ru	4.4 hr	724	48		
--	--	--	--	--	--	¹⁰⁵ Rh	35.9 hr	319	19		
Rh	¹⁰³ Rh	100	139	1100	7.9	^{106m} Rh	4.4 min	51	47		
Pd	¹⁰⁸ Pd	26.8	12	186	16	¹⁰⁹ Pd	13.5 hr	88	5		
Ag	¹⁰⁹ Ag	48.6	4.7	55	12	^{110m} Ag	255 d	658	96		
Cd	¹¹⁴ Cd	28.9	0.3	20	67	¹¹⁵ Cd	53.5 hr	528	26		
--	--	--	--	--	--	^{115m} In	4.5 hr	336	50		
In	¹¹³ In	4.3	7.8	~ 180	~ 20	^{114m} In	50 d	190	17		
--	¹¹³ In	95.7	161	2600	16	^{116m} In	54 min	1293	80		
Sn	¹¹² Sn	0.95	0.71	27	38	¹¹³ Sn	117 d	In x rays	--		
--	--	--	--	--	--	^{113m} In	100 min	391	64		
--	¹¹⁸ Sn	14.2	0.006	0.49	82	^{117m} Sn	14.0 d	158	87		
--	¹²² Sn	4.7	0.15	0.83	5.5	^{123m} Sn	40 min	160	84		
--	¹³⁴ Sn	6	0.14	9	64	^{125m} Sn	9.6 min	332	97		
Sb	¹²¹ Sb	57.3	6.2	180	29	¹²² Sb	2.8 d	564	66		
--	¹²³ Sb	42.7	4	120	30	¹²⁴ Sb	60 d	1691	50		
Te	¹³⁰ Te	34.5	0.2	0.4	2	¹³¹ Te	25 min	150	68		
--	--	--	--	--	--	¹³¹ I	8.0 d	365	82		

TABLE 4. (continued)

Element			Neutron activation cross section (barn)				Radionuclide formed			Most useful (-ray)	
Element	Stable isotope	Abundance (%)	Thermal (F)	Epithermal (I)	1 / F	Radionuclide formed	Half-Life	Energy (keV)	Abundance (%)		
--	--	--	30	465	16	¹³⁴ Cs	5.05 yr	797	99		
Ba	¹³⁰ Ba	0.12	11	270	25	¹³¹ Ba	12.0 d	496	48		
--	¹³⁴ Ba	2.6	0.16	24	150	^{135m} Ba	28.7 hr	268	16		
--	¹³⁸ Ba	70.4	0.35	0.3	0.9	¹³⁹ Ba	83 min	166	23		
La	¹³⁹ La	100	9	11	1.2	¹⁴⁰ La	40.2 hr	1597	96		
Ce	¹⁴⁰ Ce	88.5	0.58	0.49	0.8	¹⁴¹ Ce	33 d	146	48		
--	¹⁴² Ce	11.1	1.1	1.4	1.3	¹⁴³ Ce	33 hr	293	46		
Nd	¹⁴⁶ Nd	17.2	1.4	2.8	2	¹⁴⁷ Nd	11.0 d	91	28		
--	¹⁴⁸ Nd	5.7	2.5	17	6.8	¹⁴⁹ Nd	1.9 hr	211	27		
--	¹⁵⁰ Nd	5.6	1.3	14	11	¹⁵¹ Nd	12 min	116	40		
--	--	--	--	--	--	¹⁵¹ Pm	28 hr	340	21		
Sm	¹⁵² Sm	26.7	210	2900	14	¹⁵³ Sm	47 hr	103	28		
--	¹⁵⁴ Sm	22.6	5.5	27	4.9	¹⁵⁵ Sm	22 min	104	73		
Eu	¹⁵¹ Eu	47.8	2800	11400	4.1	^{152m} Eu	9.3 hr	963	12		
--	--	--	5300	3800	0.7	¹⁵² Eu	12.5 yr	1408	22		
--	¹⁵³ Eu	52.2	400	1500	3.8	¹⁵⁴ Eu	16 yr	1274	37		
Gd	¹⁵² Gd	0.21	1100	3000	2.7	¹⁵³ Gd	242 d	98	55		
--	¹⁵⁸ Gd	24.8	3.5	80	23	¹⁵⁹ Gd	18 hr	363	9		
Tb	¹⁵⁹ Tb	100	25	400	16	¹⁶⁰ Tb	72 d	299	30		
Dy	¹⁶⁴ Dy	28.1	2100	--	--	^{165m} Dy	1.26 min	Dy x rays	--		
--	--	--	2600	800	0.3	¹⁶⁵ Dy	2.32 hr	95	4		

TABLE 4. (continued)

Element	Neutron activation cross section (barn)			Most useful γ -ray					
	Stable isotope	Abundance (%)	Thermal (F)	Epithermal (I)	1 / F	Radionuclide formed	Half-Life	Energy (keV)	Abundance (%)
Tm	¹⁸⁷ Tm	100	106	1700	16	¹⁷⁰ Tm	130 d	84	3.3
Yb	¹⁶⁸ Yb	0.14	3200	25000	7.8	¹⁸⁸ Yb	31 d	198	35
--	¹⁷⁴ Yb	31.7	65	31	0.48	¹⁷⁵ Yb	4.2 d	396	6.0
--	¹⁷⁶ Yb	12.6	5.5	7	1.3	¹⁷⁷ Yb	1.9 hr	150	16
Lu	¹⁷⁵ Lu	37.4	18	600	33	^{176m} Lu	3.7 hr	88	10
--	¹⁷⁶ Lu	2.6	2050	~1100	~0.5	¹⁷⁷ Lu	6.7 d	208	6.1
Hf	¹⁷⁹ Hf	27.2	52	--	--	^{179m} Hf	18.6 s	214	94
--	¹⁸⁰ Hf	35.2	12.6	28	2.2	¹⁸¹ Hf	43 d	482	81
Ta	¹⁸¹ Ta	100	22	700	32	¹⁸² Ta	115 d	1222	34
W	¹⁸⁶ W	28.4	37	420	11	¹⁸⁷ W	24.0 hr	686	27
Re	¹⁸⁵ Re	37.1	110	1700	15	¹⁸⁶ Re	3.7 d	137	9
--	¹⁸⁷ Re	62.9	75	310	4.1	¹⁸⁸ Re	16.8 hr	155	10
Os	¹⁹⁰ Os	26.4	16	39	2.4	¹⁹¹ Os	15 d	129	25
Ir	¹⁹¹ Ir	3.85	925	4000	4.3	¹⁹² Ir	74 d	468	49
--	¹⁹³ Ir	61.5	110	1390	13	¹⁹⁴ Ir	17.4 hr	328	10
Pt	¹⁹⁵ Pt	25.2	0.8	~6	~8	¹⁹⁷ Pt	19 hr	77	20
--	¹⁹⁸ Pt	7.2	3.7	53	14	¹⁹⁹ Pt	30 min	543	24
--	--	--	--	--	--	¹⁹⁹ Au	3.15 d	158	37
Au	¹⁹⁷ Au	100	98.8	1550	16	¹⁹⁸ Au	2.69 d	412	95
Hg	¹⁹⁹ Hg	0.15	3000	~1220	~0.4	¹⁹⁷ Hg	2.70 d	78	18
--	²⁰² Hg	29.8	4.9	4.3	0.9	²⁰³ Hg	47d	279	77

TABLE 4. (continued)

Element	Stable isotope		Abundance (%)	Neutron activation cross section (barn)			Most useful (-ray)			
				Thermal (F)	Epithermal (I)	1 / F	Radionuclide formed	Half-Life	Energy (keV)	Abundance (%)
U	²³⁵ U		0.72	580 ^d	275 ^d	0.5	Fission products	Various	Various	--
--	²³⁸ U		99.3	2.72	280	103	²³⁹ U	23.5 min	75	51
--	--	--	--	--	--	--	²³⁸ Np	2.35 d	278	14

TABLE 5. NUCLEAR PARAMETERS OF LEPD - SENSITIVE ELEMENTS

Element	Isotope	Abundance	Half-life	Nuclear cross section	Low-energy photon energies (keV)
Selenium	⁷⁵ Se	0.87	120.0 days	30.0	10.7, 66.3, 96.7, 121.1, 136.0
Selenium	^{79m} Se	23.52	3.9 min.	0.4	95.9
Mercury	¹⁹⁷ Hg	0.14	65.0 hr.	905.0	66.98, 68.79, 77.97
Cadmium	¹¹⁵ Cd	28.86	53.0 hr.	1.2	24.2
Cobalt	^{60m} Co	100.00	10.5 min.	37	58.5
Platinum	¹⁹⁷ Pt	25.30	20.0 hr.	1.0.0	66.8, 75.7, 99.0, 129.7
Palladium	^{109m} Pd	26.71	5.0 min.	12.2	188.9
Barium	¹³⁹ Ba	71.66	82.9 min.	0.4	36.4, 165.8
Tungsten	¹⁸⁷ W	28.41	24.0 hr.	40.0	69.3, 71.2, 72.3, 134.3
Germanium	⁷⁵ Ge	36.54	82.0 min.	0.5	198.6, 264.6
Cesium	^{134m} Cs	100.00	2.9 hr.	30.6	31.0, 35.0, 127.4
Tin	¹²³ Sn	4.72	39.4 min.	0.2	160.2
Antimony	^{122m} Sb	57.25	3.5 min.	6.0	61.6, 76.3
Europium	^{152m} Eu	47.82	9.3 hr.	8730.0	40, 122
Samarium	¹⁵³ Sm	26.72	46.7 hr.	210.0	41, 70, 103
Gadolinium	¹⁵⁹ Gd	24.87	18.0 hr.	3.4	44
Terbium	¹⁶⁰ Tb	100.00	73.0 days	46.0	46, 87
Dysprosium	¹⁶⁵ Dy	28.18	2.3 hr.	700.0	47, 98
Holmium	¹⁶⁶ Ho	100.00	27.0 hr.	64.0	49, 81
Erbium	¹⁷¹ Er	4.88	7.5 hr.	9.0	41, 118
Ytterbium	¹⁶⁹ Yb	0.14	32.0 days	11,000.0	54, 190
Ytterbium	¹⁷⁵ Yb	31.84	4.2 days	9.0	54
Lutetium	¹⁷⁷ Lu	2.59	6.8 days	2100.0	56, 88, 113
Palladium	¹⁰⁹ Pd	26.70	13.6 hr.	12.2	22, 41, 88, 104

TABLE 6. RADIONUCLIDE STANDARDS

SHORT - LIVED RADIONUCLIDES STANDARDS		MEDIUM - LIVED RADIONUCLIDES STANDARDS		LONG - LIVED RADIONUCLIDE STANDARDS	
ELEMENT	CONCENTRATION (µgrams)	ELEMENT	CONCENTRATION (µgrams)	ELEMENT	CONCENTRATION (µgrams)
Titanium	250.0	Mercury	10.0	Cerium	25.0
Iodine	250.0	Samarium	0.50	Calcium	500.0
Manganese	1.00	Tungsten	5.00	Lutetium	0.02
Magnesium	500.0	Molybdenum	50.0	Selenium	25.0
Copper	500.0	Uranium	5.00	Terbium	5.00
Sodium	20.0	Lanthanum	5.00	Thorium	10.0
Vanadium	5.00	Cadmium	50.0	Chromium	25.0
Potassium	500.0	Gold	1.00	Europium	0.010
Chlorine	500.0	Arsenic	10.0	Ytterbium	1.00
Aluminum	250.0	Antimony	5.00	Hafnium	10.0
		Bromine	1.00	Barium	500.0
		Sodium	20.0	Strontium	200.0
		Potassium	500.0	Neodymium	25.0
		Platinum	10.0	Silver	0.500
				Zirconium	250.0
				Cesium	25.0
				Nickel	250.0
				Scandium	2.50
				Rubidium	250.0
				Iron	12,500.0
				Zinc	100.0
				Cobalt	1.00
				Tantalum	25.0

TABLE 7. GOLD FLUX MONITOR COMPARISON¹

Flux Monitor Run	Date	Net Counts at 0.411 MeV Au-198 Gamma Peak
Flux Run 1	January	58,266 ± 0.4%
Flux Run 2	February	58,397 ± 0.4%
Flux Run 3	March	58,402 ± 0.4%
Flux Run 4	April	58,122 ± 0.4%
Flux Run 5	May	58,316 ± 0.4%
Flux Run 6	June	58,105 ± 0.4%
Flux Run 7	July	58,247 ± 0.4%
Flux Run 8	August	58,399 ± 0.4%
Flux Run 9	September	58,518 ± 0.4%
Flux Run 10	October	58,175 ± 0.4%
Flux Run 11	November	58,348 ± 0.4%
Flux Run 12	December	58,433 ± 0.4%
	Average	58,310 ± 208

¹ Information in this table is an indication of the potential flux variation from one reactor irradiation to another throughout the sample year using duplicate reactor parameters. There is a 0.4% error is associated with the counting statistics.

TABLE 8. EXAMPLE OF 38% HPGE DETECTOR
EFFICIENCY STABILITY STUDIES¹

Date	609.3 KeV Gamma Counts
July 27, 1994	26,758 ± 0.6%
July 28, 1994	26,523 ± 0.6%
July 29, 1994	26,840 ± 0.6%
July 30, 1994	26,635 ± 0.6%
July 31, 1994	26,402 ± 0.6%
June 30, 1994	26,677 ± 0.6%
May 16, 1994	26,770 ± 0.6%
April 18, 1994	26,554 ± 0.6%
March 14, 1994	26,726 ± 0.6%
February 7, 1994	26,836 ± 0.6%
January 10, 1994	26,786 ± 0.6%
December 31, 1993	26,554 ± 0.6%
November 27, 1993	26,766 ± 0.6%
October 21, 1993	26,670 ± 0.6%
September 28, 1993	26,749 ± 0.6%
August 23, 1993	26,741 ± 0.6%
AVERAGE	26,687 ± 285

¹ Based on 300 second count of Ra-226 Standard Source in a fixed geometry.

TABLE 9. I, Br, Cl STANDARD COMPARISON¹

Standard	I	Br	Cl
EPA Standard 1	71326	28314	18220
EPA Standard 2	71538	28630	18357
EPA Standard 3	71105	28127	18201
EPA Standard 17	71250	28230	18488
EPA Standard 18	71612	29450	18245
EPA Standard 19	71427	28421	18119
EPA Standard 37	71582	28819	17860
EPA Standard 38	71851	28844	18092
EPA Standard 39	71841	28560	18031
EPA Standard 41	72124	29006	18545
EPA Standard 42	72831	28861	18344
EPA Standard 43	72199	28796	18566
EPA Standard 71	72828	28860	18339
EPA Standard 72	71868	28723	18316
EPA Standard 73	72017	29495	18221

- ¹ (1) These are integrated gamma ray photopeak net counts for the 442.0 MeV I-128 gamma ray, the 617.0 MeV Br-80 gamma ray, and the 1642.0 MeV Cl-38 gamma ray of typical standards used for short-lived analyses.
- (2) Each standard is composed of 200 µgrams Cl (200 lambda), 25 µgrams Br (25 lambda), and 25 µgrams I (25 lambda) for a total volume of 250 lambda (0.25 ml).
- (3) Each standard represents a separate 3 minute pneumatic rabbit irradiation at 1×10^{13} n/cm²-sec, 21 minute decay, and 500 sec. count on a 35 percent GeLi Gamma Spectrometry System.
- (4) Standards 1 through 3 were prepared as fresh stock solutions.
- (5) Standards 17 through 19 were prepared as fresh stock solutions.
- (6) Standards 37 through 39 were prepared as fresh stock solutions.
- (7) Standards 41 through 43 and 71 through 73 were prepared as fresh stock solutions.
- (8) This table provides information on the sum of errors associated with (a) weighing, (b) pipetting, (c) individual reactor irradiations, (d) counting statistics, and (e) detector efficiency and resolution stability over a period of 1 year.

TABLE 10. NAA OF TRACE ELEMENTS IN NIST 1632¹
(μgrams/gram)

The value in brackets in the certified or best known value for this element in this Standard Reference Material.

Element	NAA Value	NIST Certified Value
Titanium	944.17	(960)
Tin	15.0	(10.1)
Iodine	2.713	(2.88)
Manganese	38.957	(40)
Magnesium	1625.16	(1600)
Copper	< 20.0	(18)
Silicon	31,857.35	(33,800)
Vanadium	34.771	(35)
Chlorine	947.63	(962)
Aluminum	18,100.96	(17,800)
Mercury	0.141	(0.12)
Samarium	1.637	(1.66)
Tungsten	< 3.0	-----
Molybdenum	3.867	(3.82)
Uranium	1.414	(1.4)
Lanthanum	10.389	(10.5)
Cadmium	< 0.25	-----
Gold	< 0.00001	-----
Arsenic	5.894	(5.9)
Antimony	3.629	(3.6)
Bromine	17.787	(17.7)

Element	NAA Value	NIST Certified Value
Sodium	387.95	(380)
Potassium	2854.84	(2900)
Cerium	20.06	(19.6)
Calcium	4225.14	(4100)
Lutetium	0.13	(0.13)
Europium	0.343	(0.34)
Selenium	2.947	(2.9)
Thorium	3.1	(3.1)
Chromium	20.287	(20.2)
Ytterbium	0.788	(0.78)
Neodymium	3.0	-----
Barium	351.28	(342)
Cesium	1.52	(1.52)
Silver	< 0.09	(0.056)
Nickel	< 20.0	(15)
Scandium	3.881	(3.86)
Rubidium	21.441	(21.1)
Iron	8741.63	(8700)
Zinc	34.256	(37)
Cobalt	5.832	(5.78)

TABLE 11. NAA OF SE IN NIST SRM 1643 - A WATER
(nanograms Se/ml water)

Report No.	NIST Certified Value	NAA Measured Value
333260	11.0 ± 1.0	11.092
333227	11.0 ± 1.0	10.907
333227	11.0 ± 1.0	11.116
333233	11.0 ± 1.0	11.202
333233	11.0 ± 1.0	10.591
333233	11.0 ± 1.0	11.000
333233	11.0 ± 1.0	10.967
333226	11.0 ± 1.0	11.186
333226	11.0 ± 1.0	11.311
315670	11.0 ± 1.0	10.550
315670	11.0 ± 1.0	11.223
315612	11.0 ± 1.0	10.864
315612	11.0 ± 1.0	11.142

TABLE 12. QA NAA ANALYSES OF NIST SRM 1566 OYSTER
(µgrams element/gram SRM)

Report #	Cd	As	Zn	Selenium	Copper
NIST Certified Values	3.5 ± 0.4	13.4 ± 1.9	852.0 ± 14.0	2.1 ± 0.5	63.0 ± 3.5
333266	3.351	13.618	842.51	2.278	64.729
333266	3.301	13.512	846.25	2.194	63.686
333296	3.477	13.342	8842.47	2.041	61.149
348760	3.613	13.371	843.43	2.044	63.617
348766	3.485	14.324	842.26	2.149	62.103
348766	3.528	13.665	857.52	2.083	63.152
348778	3.413	12.188	854.76	2.137	66.901
348114	3.496	12.745	842.92	1.960	64.119
348115	3.67	12.978	863.47	2.331	63.253
348115	3.31	14.185	846.12	2.099	61.317
348112	3.232	12.439	863.92	2.077	61.749
315668	3.867	13.729	855.3	2.004	60.355
315668	3.693	13.023	856.11	2.081	63.301
315668	3.29	13.026	848.85	2.217	61.972
315668	3.877	13.193	849.47	2.103	63.580
315668	3.421	13.178	867.33	2.072	61.753
315668	3.422	13.288	845.83	2.085	60.981
315648	3.565	13.006	847.01	2.095	64.620
315685	3.383	13.317	860.01	2.234	61.347
315695	3.796	13.407	860.85	2.064	65.079
315685	3.226	13.357	844.43	2.111	62.318

TABLE 13. QA NAA ANALYSES OF NIST SRM RM-50 TUNA
(µgrams element/gram SRM)

Report No.	Mercury	Arsenic	Selenium	Zinc
NIST Certified Values	0.95 ± 0.1	3.3 ± 0.4	3.6 ± 0.4	13.6 ± 1.0
333215	1.003	3.407	3.646	12.752
331215	0.086	3.494	3.852	13.876
333266	0.919	3.278	3.537	13.417
333266	0.903	3.269	3.662	13.876
333296	0.996	3.509	3.578	13.553
333296	0.988	3.164	3.794	14.172
348766	1.005	3.365	3.864	14.021
348766	0.946	3.278	3.828	14.487
348766	0.89	3.497	3.461	14.134
348766	1.019	3.299	3.669	13.731
348766	0.957	3.256	3.627	12.821
348766	0.983	3.56	3.554	12.878
348766	1.002	3.482	3.49	13.925
348778	0.923	3.412	3.483	14.552
348115	0.982	3.544	3.994	14.238
315685	0.933	3.699	3.726	14.008
315685	----	3.346	3.514	13.872
348760	0.995	3.348	3.519	14.347
348760	0.942	3.256	3.731	13.901
348760	1.134	3.278	3.695	13.335
348760	0.974	3.544	3.9	12.843
348760	0.956	3.525	3.59	12.725

TABLE 14. (continued)

CANADA	NATIONAL RESEARCH COUNCIL CANADA
GRS-1 Gold Tailings	
UTS-2 Uranium Tailings	
UTS-4 Uranium Tailings	
FER-4 Iron Formation Rock	PACS-1 Marine Sediments
FER-1 Iron Formation Rock	DOLT-1 Dogfish Liver
FER-2 Iron Formation Rock	DORM-1 Dogfish Muscle
FER-3 Iron Formation Rock	TORT-1 Lobster
MP-2 W-Mo Ore	
DL-1A Uranium-Thorium Ore	
DH-1A Uranium-Thorium Ore	
KC-1 Zn, Pb, Sn, and Ag	
PTC-1 Flotation Rocks	
SO-1 Reference Soil	
MA-1 Gold Ore	
SCH-1 Iron Ore	
PD-1 Non Ferrous Dust	
GFETC 82-3664 Coal	
GFETC 83-0008 Coal	
SO-2 Reference Soil	
SO-3 Reference Soil	
CLV-1 Vegetation	
EASTMAN KODAK	
TEG-50-C Gelatin	
TEG-50-B Gelatin	
USGS	
W2 Diabase Soil	
BHVO-1 Basalt	
106-A Monazite Sand	
107-A Monazite Sand	
110-A Monazite Sand	
AGV-1 Andesite	
C20280 Coal	
CLB-1 Coal	

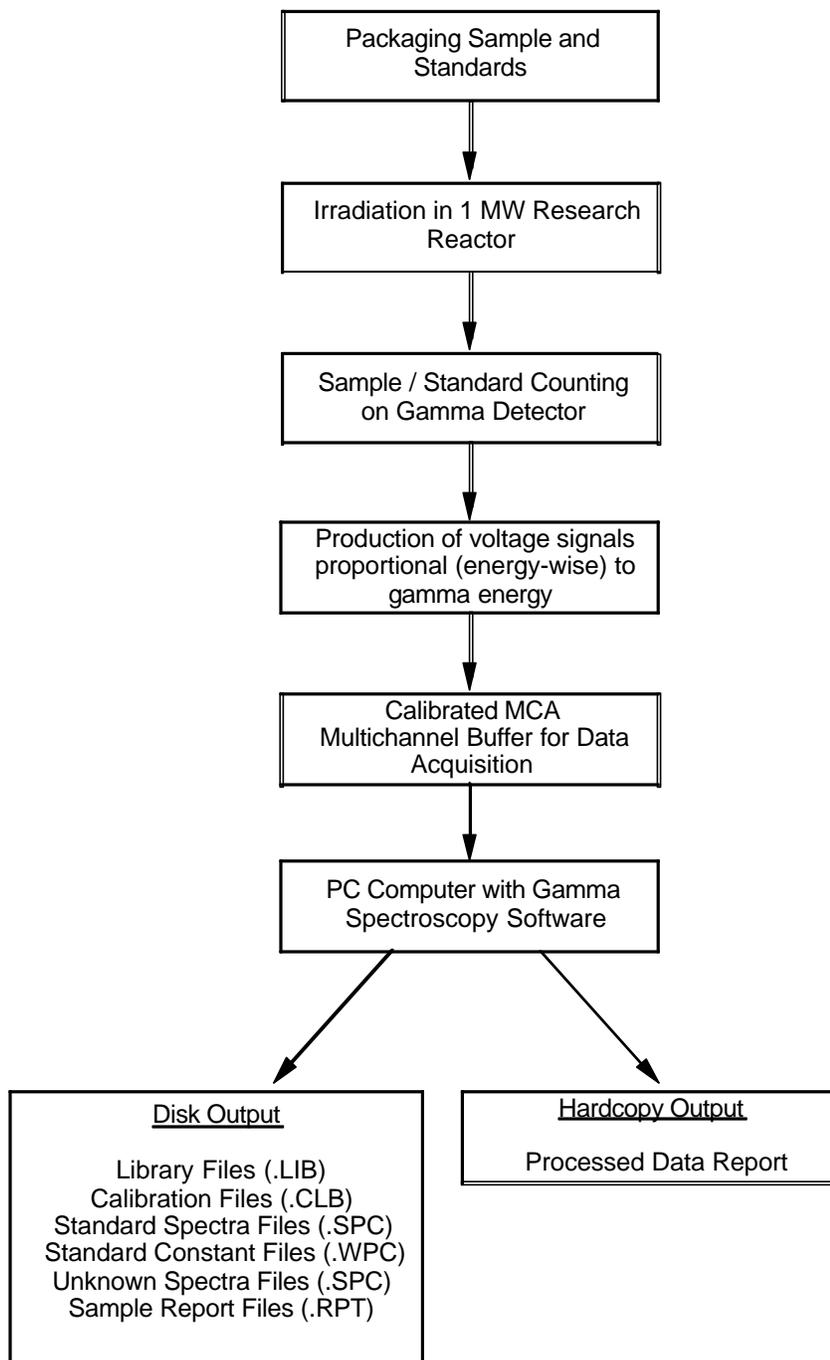
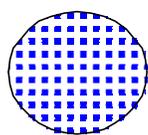


Figure 1. Neutron Activation Analysis process.

FOLDING PROCESS



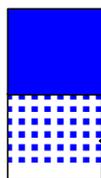
Filter



Filter folded in half



Filter quartered



Irradiation vial with
Inserted filter

Figure 2. Air filter folding process.

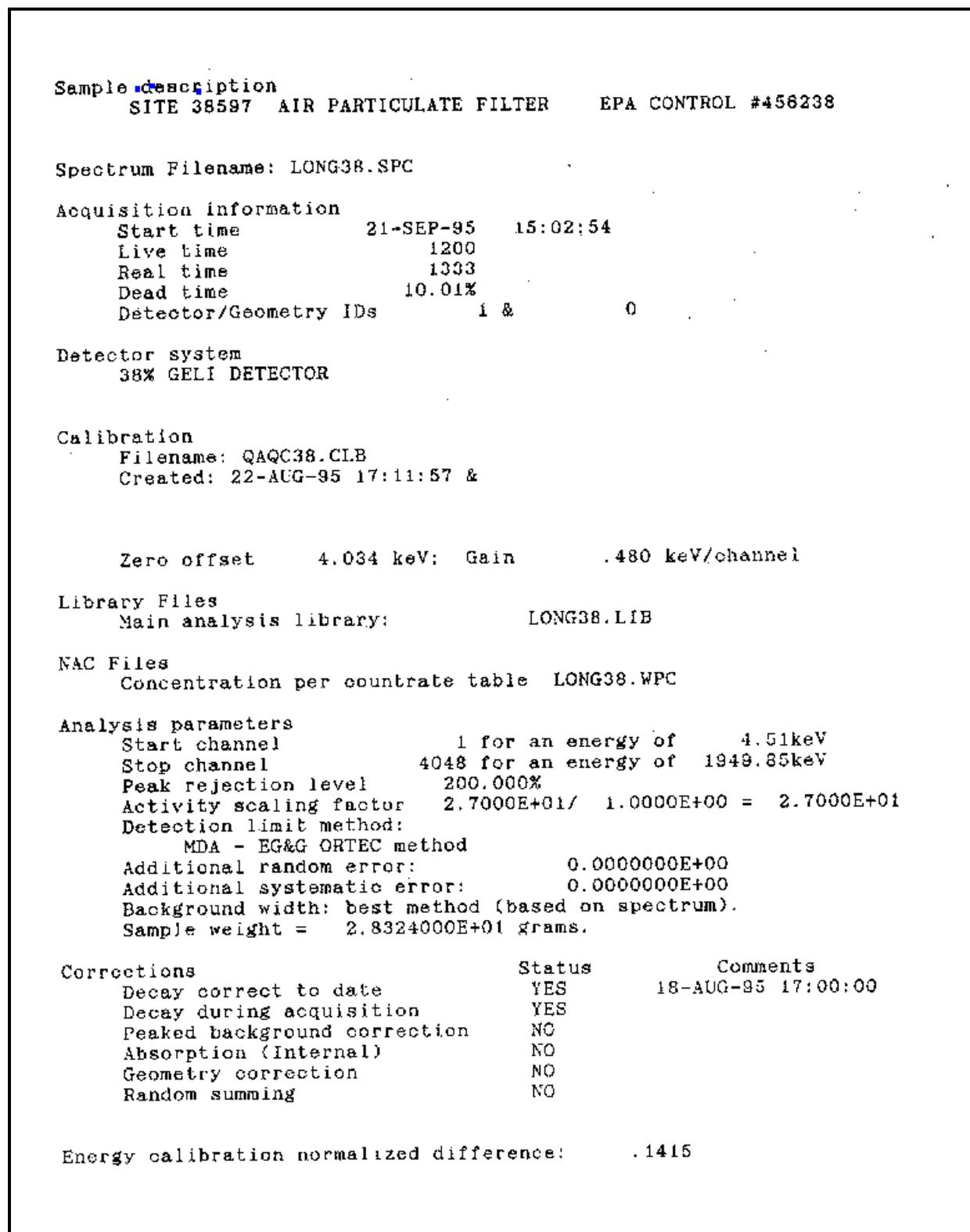


Figure 3. NAA sample output data report.

CHANNEL	ENERGY	BACKGROUND	NET AREA	CNTS/SEC	UNCERT	FWHM	SUSPECTED
820.28	398.30	8830.	155.	.13	90.10	.810	- sM
825.82	400.88	8333.	443.	.37	48.61	1.193	Se-75 sM
846.20	410.77	8287.	380.	.30	42.31	1.271	Eu-152 s
856.12	415.54	6749.	613.	.51	23.68	1.495	Eu-152 s
915.28	444.00	6511.	495.	.41	24.87	1.772	Eu-152 s
926.27	449.29	8670.	245.	.20	66.03	1.308	-
981.57	475.89	5907.	196.	.16	64.32	.923	Bi-214 s
1013.96	491.46	3699.	61.	.05	111.90	.832	- sM
1126.55	545.60	8446.	155.	.13	98.76	.992	Cs-138 s
1138.26	551.23	6945.	160.	.13	78.38	1.381	W-187 s
1146.70	555.29	7829.	179.	.15	78.66	1.058	Y-91M s
1163.94	563.57	12028.	817.	.88	30.24	2.078	As-76 s
1175.80	569.27	8001.	1054.	.88	15.24	1.350	Cs-134 s
1199.96	580.89	6782.	68.	.06	149.46	1.004	Pb-214 s
1248.80	604.37	15695.	8213.	6.84	3.42	1.588	Cs-134 s
1271.46	615.26	7944.	484.	.39	29.18	1.078	Ru-106 s
1280.63	619.67	10441.	458.	.38	45.08	1.682	Br-82 s
1333.00	644.84	11478.	149.	.12	183.71	.000	- s
1350.89	653.44	6897.	91.	.08	108.61	.501	Sr-91 sM
1485.47	718.11	11230.	491.	.41	42.04	1.066	Bi-214 s
1497.53	723.90	9577.	841.	.70	24.47	2.794	Zr-95 s
1584.24	765.56	5962.	577.	.48	10.49	1.548	Nb-95 s
1603.69	774.90	4523.	78.	.07	114.52	1.180	Te-131 s
1612.42	779.10	8659.	1491.	1.24	10.39	1.512	Eu-152 s
1660.63	802.26	10235.	531.	.44	34.37	1.741	Cs-134 M
1728.65	834.93	12918.	3744.	3.12	6.14	1.703	Kr-88 s
1744.88	842.72	8944.	361.	.30	54.79	.473	- s
1778.00	858.63	8645.	243.	.20	66.53	.000	- s
1821.02	879.29	15814.	1203.	1.00	26.47	1.666	Tb-160 s
1926.28	929.83	9567.	315.	.28	58.18	.726	- s
1936.61	934.79	3543.	57.	.05	139.76	.668	- s
1972.85	952.19	6615.	108.	.09	126.38	.795	- s
1997.01	963.79	7314.	302.	.25	59.19	1.508	Eu-152 s
2024.10	976.79	5416.	243.	.20	57.05	.748	- s
2160.85	1042.43	3183.	166.	.14	71.10	.753	- s
2250.63	1085.51	4158.	966.	.80	12.72	1.910	Au-198 sM
2304.71	1111.46	3160.	594.	.50	15.51	1.383	Eu-152 sM
2322.99	1120.23	5271.	145640.	121.37	.30	1.799	Bi-214 M
2409.68	1161.82	2061.	121.	.10	65.62	1.116	- s
2443.29	1177.94	1785.	744.	.62	13.22	2.337	Tb-160 sM
2465.36	1188.53	2578.	613.	.51	27.75	1.948	Ta-182 s
2489.02	1199.88	1499.	135.	.11	54.31	1.118	- s
2554.40	1231.23	1387.	535.	.45	20.14	2.678	Ta-182 s
2641.15	1272.84	1718.	588.	.49	14.69	4.007	Eu-154 s
2679.09	1291.03	1910.	19017.	15.85	.90	1.960	Fe-59 s
2695.80	1299.04	1264.	162.	.14	40.04	1.259	I-133 s
2707.22	1304.52	564.	72.	.06	55.14	.745	- s
2722.40	1311.79	1399.	118.	.10	67.63	.624	- s
2764.17	1331.82	2024.	9797.	8.16	1.65	1.963	Co-60 s
2831.59	1364.14	1507.	258.	.22	41.22	1.421	- s
2905.96	1399.79	1130.	280.	.22	27.36	2.136	- s
2921.52	1407.25	1226.	1655.	1.38	5.33	1.951	Eu-152 s

Figure 3. (continued)

CHANNEL	ENERGY	BACKGROUND	NET AREA	CNTS/SEC	UNCERT	FWHM	SUSPECTED
2955.35	1423.46	285.	17.	.01	113.65	.541	- s
3002.04	1445.84	635.	79.	.07	56.53	.909	- s
3031.48	1459.94	964.	158.	.13	40.03	2.032	K-40 s
3114.49	1499.7	472.	48.	.04	69.71	.965	- s
3167.84	1525.28	504.	20.	.02	152.25	.969	K-42 s
3173.70	1528.09	812.	53.	.04	161.75	.662	- s
3284.22	1581.03	935.	104.	.09	61.32	1.464	- s
3325.20	1600.65	690.	65.	.05	81.94	.691	- s
3392.88	1633.06	549.	64.	.05	69.40	1.192	- s
3550.03	1669.16	787.	319.	.27	20.47	1.672	Sb-124 s
3625.10	1744.25	331.	23.	.02	103.11	.585	- s
3682.28	1771.62	583.	59.	.05	71.82	1.419	Co-56 s
3699.02	1779.63	535.	68.	.06	66.59	.776	- s
3962.72	1905.82	146.	17.	.01	110.16	.582	- s
3967.78	1908.24	103.	14.	.01	113.65	.751	- s

s Peak fails shape tests.
M Peak issclose to a library peak.

***** I D E N T I F I E D P E A K S U M M A R Y *****							
NUCLIDE	PEAK CHANNEL	CENTROID ENERGY	BACKGROUND COUNTS	NET AREA COUNTS	INTENSITY CTS/SEC	UNCERT 1 SIGMA	FWHM keV
CE-141	294.75	145.39	16581.	18561.	15.47	1.78	1.049
CA-SC47	323.62	159.29	10932.	137.	.11	106.06	.564s
LU-177	28.66	208.89	15220.	270.	.23	79.35	.987
SE-75	542.49	264.64	10854.	1337.	1.11	12.62	1.196
TB-160	614.62	299.36	11566.	4521.	3.77	4.98	2.548s
TH-PA233	640.52	311.82	12241.	14737.	12.28	1.57	1.237
CR-51	657.56	320.02	11009.	9420.	7.85	2.26	1.275
EU-152	707.75	344.17	10106.	7899.	6.58	2.78	1.383s
YB-175	819.38	397.87	9126.	613.	.51	31.43	1.049s
HF-181	994.66	482.18	9142.	3986.	3.32	4.45	1.360
BA-131	1025.00	496.77	9825.	526.	.44	34.22	1.331
NB- 847	1080.02	531.62	12402.	388.	.30	52.38	1.339s
AG-110M	1359.90	657.77	12933.	336.	.28	70.63	.741s
ZR-95	1566.55	757.06	8927.	334.	.28	51.31	.592s
CS-134	1647.32	775.86	8888.	4812.	4.01	4.47	1.747
NI-CO58	1677.40	810.31	7748.	474.	.39	32.52	1.694
SC-46	1841.66	869.20	17338.	175795.	146.50	.29	1.654
RE-86	2230.73	1075.96	4884.	530.	1.27	15.36	1.857s
FE-59	2278.68	1098.97	4754.	28146.	23.46	.82	1.818
ZN-65	2313.05	1115.46	5486.	904.	.75	15.69	1.214s
CO-60	2432.70	1172.86	2607.	10668.	8.89	1.4	1.699
TA-182	2533.41	1221.17	1565.	672.	.56	12.39	2.103

s Peak fails shape tests.
D Peak area deconvoluted.

Figure 3. (continued)

```

***** SUMMARY OF NUCLIDES IN SAMPLE *****
TIME OF COUNT   TIME CORRRECTED   UNCERTAINTY   1 SIGMA
NUCLIDE         ACTIVITY             ACTIVITY             COUNTING
                PCI/FILTER        PCI/FILTER
+-----+-----+-----+-----+-----+-----+-----+-----+
CE-141          5.7446E+01           1.1842E+02           1.78%
CA-SC47         2.9450E-01           2.7921E+02           108.06%
LU-177 <        9.29E-02             3.02E+00
SE-75           6.7286E-01           8.1717E-01           12.62%
TB-160 <        3.02E-02             4.17E-02
CR-84233        3.4579E+00           9.3309E+00           2.56%
EU-152 #        1.6722E+00           1.6810E+00           2.78%
YB-176 <        1.44E-02             3.84E+00
HF-181          8.7521E-01           1.4827E+00           4.46%
SR-85 <         4.1737E-02           3.2298E+02           34.22%
ND-147 #        1.7114E-01           1.4340E+00           52.29%
AG-110M#        1.1270E-01           1.2367E-01           70.63%
ZR-95           6.3816E-01           9.1826E-01           51.31%
NS-CO58         1.0692E+00           1.3090E+00           32.62%
SC-46           3.1370E+02           4.1516E+02           .29%
RB-86 #         1.8377E+00           6.4784E+00           15.36%
FE-59           3.2557E+01           5.4832E+01           .82%
ZN-66 <         1.2133E+02           1.2263E+02           1.44%
TA-182          6.1669E-01           1.0018E+00           12.39%

# All peaks for activity calculation had bad shape.
----- SUMMARY -----
TOTAL AACTIVITY ( 3.9 to 1946.6 keV) 9.2716130E+02 PCI/FILTER

***** SUMMARY OF DISCARDED PEAKS *****
208.40 ? LU-177      298.60 & TB-160      344.20 * EU-152      396.10 & YB-176
514.00 ? SR-85      657.40 **AG-110M    1115.40 ? ZN-65

* - Peak is too wide, but only one peak in library.
! - Peak is part of a multiplet and this area went
    negative during deconvolution.
? - Peak is too narrow.
@ - Peak is too wide aa FW25M, but ok at FWHM.
% - Peak fails sensitivity test.
$ - Peak identified, but first peak of this nuclide
    failed one or more qualification tests.
+ - Peak activity higher than counting uncertainty range.
- - Peak activity lower than counting uncertainty range.
= - Peak outside analysis energy range.
& - Calculated peak centroid is not close enough to the
    library energy centroid for positive identification.
P - Peakbackground subtraction

```

Figure 3. (continued)

SUMMARY OF NUCLIDE CONCENTRATIONS			
ELEMENT	TIME OF COUNT CONCENTRATION PPM	TIME CORRECTED CONCENTRATION PPM	UNCERTAINTY 1 SIGMA COUNTING
CERIUM	6.7245E-02	1.3862E-01	1.70%
CALCIUM	1.8274E-02	1.7325E+01	103.00%
LUTETIUM	0.0000E+00	0.0000E+00	.00%
SELENIUM	8.3820E-03	1.0180E-02	12.60%
TERBIUM	9.5249E-04	1.3144E-03	4.90%
THORIUM	9.5379E-03	2.2783E-02	1.50%
CHROMIUM	7.5336E-02	1.7550E-01	2.20%
EUROPIUM	1.6366E-03	1.6446E-03	2.70%
YTTERBIUM	2.6731E-04	7.1331E-02	31.40%
HALFNIUM	3.6694E-03	6.2163E-03	4.40%
BARIUM	9.9312E-02	7.6714E-01	34.20%
STRONTIUM	2.2194E-01	3.2047E-01	39.30%
NEODYMIUM	7.6179E-03	6.3831E-02	52.20%
SILVER	1.0487E-02	1.1509E-02	70.60%
ZIRCONIUM	7.5958E-02	1.0906E-01	51.30%
CESIUM	6.6270E-03	6.8363E-03	4.40%
NICKEL	3.1279E-02	7.1309E-02	32.50%
SCANDIUM	1.5329E-02	2.0287E-02	.20%
RUBIDIUM	6.7420E-02	2.3767E-01	15.30%
IRON	1.8756E+01	3.1590E+01	.80%
ZINC	4.9523E-02	5.4510E-02	15.60%
COBALT	2.1168E-02	2.1734E-02	1.40%
TANTALUM	9.8842E-04	1.2136E-03	12.30%

***** SUMMARY OF DISCARDED PEAKS *****

208.40 ? LU-177

- * - Peak is too wide, but only one peak in library.
- ! - Peak is part of a multiplet and this area went negative during deconvolution.
- ? - Peak is too narrow.
- ⊙ - Peak is too wide at FW25M, but ok at FWHM.
- % - Peak fails sensitivity test.
- \$ - Peak identified, but first peak of this nuclide failed one or more qualification tests.
- + - Peak activity higher than counting uncertainty range.
- - Peak activity lower than counting uncertainty range.
- = - Peak outside analysis energy range.
- & - Calculated peak centroid is not close enough to the library energy centroid for positive identification.
- P - Peakbackground subtraction

Figure 3. (continued)